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Vapor Pressures and Sublimation Enthalpies of Cadmium Difluoride and Zinc Difluoride by the Torsion-Effusion Method

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The vapor pressures over solid CdF_2 and ZnF_2 were measured by the torsion-effusion method. The temperature dependencies of the vapor pressures of these compounds were found to fit the following equations: $\log(p/\text{kPa}) = (9.55 \pm 0.30) - (13920 \pm 300)(T/\text{K})$ (from (962 to 1149) K), and $\log(p/\text{kPa}) = (9.79 \pm 0.40) - (12510 \pm 400)(T/\text{K})$ (from (846 to 1047) K) for CdF_2 and ZnF_2, respectively. Treating these vapor pressures by the second- and third-law methods, the standard sublimation enthalpies $\Delta_{\text{sub}}H°(298\text{ K}) = (285 \pm 10)\text{ kJ}\cdot\text{mol}^{-1}$ and $(257 \pm 8)\text{ kJ}\cdot\text{mol}^{-1}$ and entropies $\Delta_{\text{sub}}S°(298\text{ K}) = (163 \pm 6)\text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$ and $(169 \pm 8)\text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$ for CdF_2 and ZnF_2, respectively, were calculated.

Introduction

The vapor pressures above both molten CdF_2 and ZnF_2 were measured at high temperatures by the "boiling-point" method[1] over the ranges from (1638 to 2023) K and (1429 to 1738) K for CdF_2 and ZnF_2, respectively. The vapor pressures above solid CdF_2 were measured by the Knudsen and Langmuir methods,[2] and those above solid ZnF_2 by only the Knudsen method.[3] By the second- and third-law treatment of the vapor pressures, the sublimation enthalpies and entropies of these compounds were derived. Apparently no other sublimation thermodynamic data were found in the literature so that the aim of this work was to measure new sets of the absolute vapor pressures of solid CdF_2 and ZnF_2 by the torsion method and from these values to determine their standard sublimation thermodynamic values.

Experimental Section

Four different samples of CdF_2 supplied by Alfa Aesar with a nominal purity of 99.99 % (lot: S95130) and two ZnF_2 samples, with a nominal purity of 99 %, supplied by Aldrich were employed in this study. The vapor pressures of the compounds were measured using the torsion assembly described in detail in a previous work. [4] A conventional torsion cell (A), machined by graphite at practically zero porosity, and another one (B) machined by graphite having a platinum foil as liner with effusion holes having different diameters ((0.6 and 1.5) mm for cell A and B, respectively) were used. The torsion constants of these cells, necessary to convert the measured torsion angles into pressure values, were determined by vaporizing very pure lead, a standard element, the vapor pressures of which are well-known[5] and comparable with those of the studied compounds and therefore measurable in comparable temperature ranges. Checks of these constants for both cells obtained in several experiments carried out before and/or after each vaporization run showed that they were well reproducible ranging within about 10 % of their mean values. In any case, in each vaporization run the absolute vapor pressures were calculated using the torsion constant value obtained in the calibration run carried before and/or after the vaporization run. Considering negligible the errors in the torsion angle measurements, the uncertainty of the torsion constant value of both cells produced a displacement of the logarithm of the absolute pressure values decidedly no larger than about ± 0.03 to 0.04. Several second-law vaporization enthalpy values of lead were also determined from the slopes of the log α versus $1/T$ equations, where α are the torsion angles (see Figure 1) obtained in the calibration runs. The excellent agreement of the obtained $\Delta H°(T)$ values themselves and their average values, $(183 \pm 3)\text{ kJ}\cdot\text{mol}^{-1}$ and $(186 \pm 4)\text{ kJ}\cdot\text{mol}^{-1}$ for the cell A and B, respectively, with that selected by Hultgren et al.[5] at 1000 K, approximated as the middle of the experimental temperature ranges, $[\Delta_{\text{sub}}H°(1000\text{ K}) = 184\text{ kJ}\cdot\text{mol}^{-1}]$, shows that in the operative conditions the thermodynamic equilibrium between the condensed and vapor phase in the used cells was attained and that the measured temperatures were fairly reliable with an uncertainty that we believe should not exceed ± 2 K.

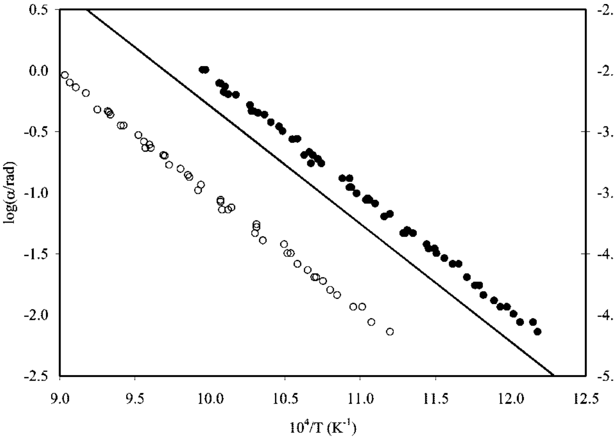

Figure 1. Torsion angles measured in several calibration runs vaporizing lead by ○, the cell A; and ●, the cell B (with Pt liner). The line represents the selected vapor pressure of lead.[5]

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Table 1. Torsion Vapor Pressures of CdF_2

Cell A									
run 4		run 6		run 9		run 11		run 13	
T/K	−log (p/kPa)	T/K	−log (p/kPa)	T/K	−log (p/kPa)	T/K	−log (p/kPa)	T/K	−log (p/kPa)
1027	4.04	1032	4.04	1033	4.04	1014	4.24	1017	4.24
1038	3.94	1040	3.87	1043	3.87	1026	4.10	1025	4.17
1048	3.80	1050	3.74	1055	3.74	1032	3.99	1036	3.99
1057	3.69	1059	3.64	1066	3.56	1043	3.86	1046	3.86
1068	3.53	1068	3.50	1077	3.44	1054	3.69	1057	3.69
1079	3.39	1078	3.39	1087	3.30	1064	3.56	1068	3.56
1090	3.26	1088	3.26	1096	3.20	1075	3.41	1079	3.47
1102	3.14	1098	3.14	1107	3.09	1087	3.30	1089	3.34
1114	3.00	1107	3.04	1118	2.96	1097	3.20	1098	3.24
1125	2.88	1117	2.93	1129	2.85	1107	3.09	1107	3.17
1136	2.75	1127	2.84	1139	2.74	1117	2.99	1118	3.00
1147	2.64	1136	2.75	1149	2.64	1126	2.89	1124	2.94
		1145	2.65			1136	2.78	1127	2.89

Cell B (Pt Liner)									
run 2		run 5		run 7		run 9		run 11	
T/K	−log (p/kPa)	T/K	−log (p/kPa)	T/K	−log (p/kPa)	T/K	−log (p/kPa)	T/K	−log (p/kPa)
962	4.90	978	4.60	968	4.87	1000	4.30	1003	4.24
973	4.80	988	4.47	977	4.70	1013	4.12	1017	4.04
983	4.60	1000	4.30	984	4.57	1021	3.99	1030	3.90
993	4.46	1009	4.17	992	4.47	1031	3.87	1042	3.74
1004	4.30	1018	4.07	1001	4.33	1043	3.73	1053	3.60
1013	4.16	1028	3.93	1013	4.17	1054	3.58	1064	3.47
1022	4.06	1040	3.73	1025	3.97	1069	3.41	1075	3.32
1032	3.92	1052	3.60	1038	3.79	1080	3.30	1086	3.17
1042	3.79	1069	3.39	1049	3.64	1091	3.15	1097	3.04
1051	3.67	1079	3.28	1061	3.51	1101	3.04	1108	2.92
1060	3.56	1091	3.15	1073	3.40	1111	2.93	1118	2.80
1070	3.45	1103	3.03	1084	3.27	1120	2.77		
1079	3.34	1110	2.93	1097	3.14	1129	2.71		
1089	3.23	1115	2.87	1105	3.06	1138	2.61		
1098	3.13	1121	2.79	1114	2.96				
1107	3.04	1129	2.70	1123	2.84				
1117	2.94	1137	2.61	1135	2.71				
1127	2.84								

Cell B (Pt Liner)			
run 13		run 14	
T/K	−log (p/kPa)	T/K	−log (p/kPa)
1003	4.24	1002	4.34
1012	4.10	1016	4.17
1022	3.94	1027	4.04
1033	3.80	1046	3.80
1043	3.64	1055	3.69
1052	3.53	1064	3.56
1062	3.41	1071	3.47
1074	3.28	1082	3.34
1084	3.17	1093	3.21
1095	3.03	1104	3.09
1105	2.92	1114	2.98
1116	2.82	1125	2.86
1127	2.72	1134	2.76

In the first step of all the vaporizations of CdF_2, the vapor pressures were found not reproducible, decreasing in isothermal conditions, and only after the evaporation of some amount of the sample, with the amount depending on the sample used and varying from (2 to 8) % of its original weight, the pressures were found to be decidedly well reproducible. This was justified for the possible presence of impurities in spite of the nominal purity of the supplied samples. On the contrary, the vapor pressures of ZnF_2 were found well reproducible immediately when the samples were heated. The torsion absolute vapor pressures of purified CdF_2 and ZnF_2 obtained by using both cells and the corresponding experimental temperatures are reported in Tables 1 and 2 and in Figures 2 and 3. The log p versus $1/T$ equations reported for both compounds in Table 3 were obtained by least-squares treatment of the experimental data of each run. No evident dependence of the vapor pressures on the cell used or the diameter of the effusion hole was observed. By weighting the slope and intercept of each equation proportionally to the number of the experimental points, the following final equations for CdF_2 and ZnF_2 were selected.

$$CdF_2(s)\ \log(p/\text{kPa}) = (9.55 \pm 0.30) - (13920 \pm 300)(T/\text{K})\ \text{(from (962 to 1149) K)} \quad (1)$$

$$ZnF_2(s)\ \log(p/\text{kPa}) = (9.79 \pm 0.40) - (12510 \pm 400)(T/\text{K})\ \text{(from (846 to 1047) K)} \quad (2)$$

The small errors reported in the log p versus $1/T$ equation obtained in each run (see Table 3) are standard deviations of the experimental points from each straight line and do not include the uncertainties in the measured temperatures and in

Table 2. Torsion Vapor Pressures of ZnF_2

Cell A							
run 14		run 15		run 17		run 19	
T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$
883	4.34	887	4.34	885	4.34	888	4.34
895	4.17	900	4.17	896	4.17	898	4.17
903	4.04	911	3.94	906	4.04	909	3.99
913	3.86	921	3.74	916	3.80	919	3.80
924	3.69	930	3.60	926	3.64	928	3.69
934	3.53	940	3.47	937	3.50	939	3.50
945	3.39	951	3.32	948	3.36	948	3.36
955	3.24	961	3.18	959	3.23	958	3.24
966	3.10	972	3.04	970	3.06	968	3.12
976	2.97	982	2.92	980	2.94	978	2.98
986	2.84	991	2.80	989	2.83	989	2.86
996	2.72	1001	2.68	999	2.71	998	2.74
1006	2.61	1011	2.57	1010	2.59	1007	2.63
1017	2.49	1021	2.46	1021	2.47	1017	2.53
1027	2.39	1031	2.35	1031	2.34	1027	2.42
1036	2.28	1041	2.25	1039	2.25	1037	2.31
1046	2.17					1047	2.20

Cell A				Cell B (Pt Liner)			
run 21		run 24		run 15		run 17	
T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$
886	4.44	876	4.45	867	4.74	853	4.91
896	4.21	887	4.22	880	4.44	875	4.61
904	4.13	898	4.08	893	4.21	887	4.31
912	4.01	906	3.97	902	4.07	898	4.13
922	3.87	914	3.81	913	3.91	909	4.01
933	3.71	923	3.69	925	3.74	919	3.87
942	3.55	933	3.56	934	3.59	930	3.71
952	3.44	941	3.43	946	3.42	941	3.55
963	3.28	950	3.32	956	3.29	951	3.41
974	3.13	960	3.18	966	3.16	961	3.28
983	3.01	969	3.07	975	3.04	970	3.15
993	2.88	979	2.94	986	2.90	979	3.03
1004	2.76	988	2.83	997	2.78	989	2.90
1013	2.64	996	2.71	1008	2.66	999	2.78
1023	2.51	1004	2.61	1019	2.53	1009	2.66
1035	2.37	1013	2.50			1020	2.53
1046	2.24	1022	2.40				
		1031	2.27				
		1040	2.16				

Cell B (Pt Liner)					
run 19		run 22		run 23	
T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$
859	4.91	846	4.92	852	4.92
871	4.61	854	4.75	867	4.62
883	4.44	865	4.62	880	4.45
895	4.31	876	4.32	891	4.22
905	4.13	889	4.14	902	4.08
914	3.96	903	3.97	910	3.92
923	3.83	916	3.78	920	3.78
934	3.68	928	3.60	932	3.58
943	3.55	938	3.43	942	3.45
956	3.38	950	3.29	952	3.30
965	3.23	962	3.16	963	3.16
975	3.09	969	3.04	973	3.02
984	2.98	978	2.91	983	2.88
993	2.87	988	2.80	994	2.75
1003	2.75	998	2.67	1005	2.60
1013	2.63	1009	2.53	1016	2.46
1022	2.52				

the torsion constant value used. A more realistic appraisal of the results suggests the estimated errors reported in the selected eqs 1 and 2.

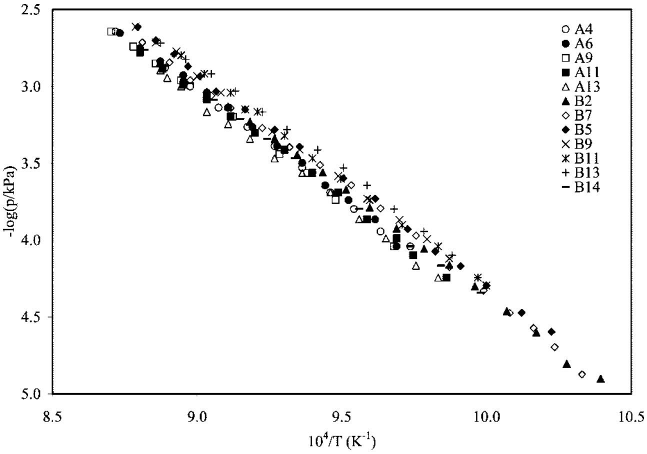

Figure 2. Torsion vapor pressures of CdF_2.

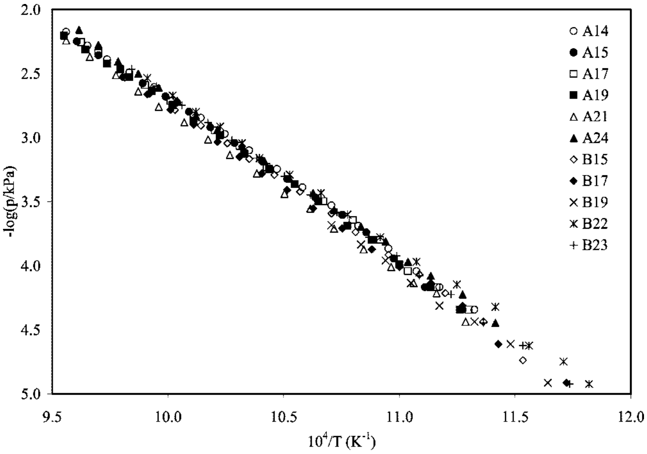

Figure 3. Torsion vapor pressures of ZnF_2.

Table 3. Temperature Dependence of Torsion Vapor Pressures and Sublimation Enthalpies of CdF_2 and ZnF_2

compound	cell	run	ΔT K	no. of points	$\log(p/\text{kPa}) = A - B/(T/\text{K})$ A^a	 B^a
CdF_2	A	A4	1027 to 1147	12	9.57 ± 0.12	13996 ± 128
	A	A6	1032 to 1145	13	9.75 ± 0.20	14175 ± 222
	A	A9	1033 to 1149	12	9.70 ± 0.17	14158 ± 184
	A	A11	1014 to 1136	13	9.41 ± 0.17	13826 ± 181
	A	A13	1017 to 1127	13	9.45 ± 0.17	13922 ± 179
	B	B2	962 to 1127	18	9.42 ± 0.11	13785 ± 114
	B	B5	978 to 1137	17	9.56 ± 0.08	13859 ± 82
	B	B7	968 to 1135	17	9.54 ± 0.16	13888 ± 166
	B	B9	1000 to 1138	14	9.53 ± 0.11	13827 ± 119
	B	B11	1003 to 1118	11	9.85 ± 0.13	14148 ± 137
	B	B13	1003 to 1127	13	9.63 ± 0.18	13874 ± 196
	B	B14	1002 to 1134	13	9.36 ± 0.08	13743 ± 86
ZnF_2	A	A14	883 to 1046	17	9.64 ± 0.11	12330 ± 102
	A	A15	887 to 1041	16	9.83 ± 0.18	12533 ± 175
	A	A17	885 to 1039	16	9.68 ± 0.15	12383 ± 140
	A	A19	888 to 1047	17	9.66 ± 0.13	12388 ± 129
	A	A21	886 to 1046	17	9.76 ± 0.07	12560 ± 65
	A	A24	876 to 1040	19	9.82 ± 0.06	12477 ± 60
	B	B15	867 to 1019	15	9.81 ± 0.16	12537 ± 150
	B	B17	853 to 1020	16	9.68 ± 0.12	12443 ± 112
	B	B19	859 to 1022	17	9.90 ± 0.11	12685 ± 104
	B	B22	846 to 1009	16	9.69 ± 0.13	12335 ± 121
	B	B23	852 to 1016	16	10.26 ± 0.07	12917 ± 64

[a] The quoted errors are standard deviations.

A. CdF_2. The comparison in Table 4 and Figure 4 of the selected eq 1 with those obtained by Knudsen and Langmuir methods by Besenbruch et al.[2] shows that our absolute vapor pressures are slightly higher. From the slope and intercept of eq 1, the second-law thermodynamic changes associated with the sublimation process at 1050 K, $\Delta_{\text{sub}}H°(1050\text{ K}) = (267 \pm$

Table 4. Comparison of the Vapor Pressures of CdF_2 and ZnF_2 with the Literature Data

				ΔT	$\log(p/\text{kPa}) = A - B/(T/\text{K})$	
compound	ref	no. of points	method	K	A	B
CdF_2	2	18	Knudsen	1092 to 1255	9.394 ± 0.015	14089 ± 80
	2	11	Langmuir	921 to 1041	9.569 ± 0.021	14341 ± 113
	this work	166	torsion	962 to 1149	9.55 ± 0.30	13920 ± 300
ZnF_2	3	40	Knudsen	901 to 1125	10.442 ± 0.071	13185 ± 72
	this work	182	torsion	846 to 1047	9.79 ± 0.40	12510 ± 400

Table 5. Third-Law Sublimation Enthalpies for CdF_2 and ZnF_2

	T	p^a	$-R \ln p$	Δfef	$\Delta_{sub}H°(298\text{ K})$
compound	K	kPa	$J\cdot K^{-1}\cdot mol^{-1}$	$J\cdot K^{-1}\cdot mol^{-1}$	$kJ\cdot mol^{-1}$
CdF_2	950	$7.95\cdot 10^{-6}$	136	176	296
	1150	$2.81\cdot 10^{-3}$	87	174	301
ZnF_2	850	$1.20\cdot 10^{-5}$	133	174	261
	1050	$7.62\cdot 10^{-3}$	79	172	263

[a] From eqs 1 and 2, respectively.

6) $kJ\cdot mol^{-1}$ and $\Delta_{sub}S°(1050\text{ K}) = (145 \pm 6)\ J\cdot K^{-1}\cdot mol^{-1}$, were calculated. These values were corrected to 298 K using the heat contents reported in the IVTANTHERMO database:[6] $\Delta_{sub}H°(298\text{ K}) = (278 \pm 6)\ kJ\cdot mol^{-1}$ and $\Delta_{sub}S°(298\text{ K}) = (163 \pm 6)\ J\cdot K^{-1}\cdot mol^{-1}$. The standard sublimation enthalpy was also calculated by third-law treatment of the experimental data at two temperatures, (950 and 1150) K, approximated extremes of the experimental temperature range. At these temperatures, the vapor pressure values were calculated from eq 1, and the free energy functions, $fef = [G°(T) - H°(298\text{ K})]/T$, for solid and gaseous phases were taken from the IVTANTHERMO database.[6] The enthalpies so calculated, and reported in Table 5, present a small temperature dependence. The average value, $\Delta_{sub}H°(298\text{ K}) = 298\ kJ\cdot mol^{-1}$, with an approximated error of 8 $kJ\cdot mol^{-1}$, is higher by 20 $kJ\cdot mol^{-1}$ than that obtained by the second-law procedure. A critical analysis of our results shows that: (i) the slopes and intercepts of all log p versus $1/T$ equations in Table 3 are decidedly in good agreement; (ii) all the temperature dependence of the torsion angles measured above pure lead in several check runs (runs A5, A10, A16, and A23 with the cell A and B6, B12, B18, and B21 with the cell B) is in excellent agreement with the temperature dependence of the vapor pressures for this compound reported in the literature[5] so that we believe that also the temperature dependence of the torsion angles measured above CdF_2, dependence from which the second-law sublimation enthalpy of this compound was calculated, could be reliable; (iii) also the absolute vapor pressures of CdF_2 could be decidedly reliable considering that these were calculated employing torsion constants obtained in runs carried out before or after the vaporization runs; (iv) although in contrast with that observed in the mass spectrometric analysis of the vapor above other fluorides,[7,8] the hypothesis of a possible partial decomposition of CdF_2(g) and/or a possible presence of a dimeric form in the vapor leads to a decrease of the CdF_2(g) partial pressure values, and this increases the third-law enthalpy value and accentuates the disagreement with the second-law result. In light of these considerations, we believe that the absolute vapor pressures above CdF_2 measured in the present work, compatible with those reported in the literature and the derived second-law sublimation enthalpy are reliable enough and that the Δfef from the database,[6] the same values as those used by Brewer et al.,[9] probably are too high. In particular, it is interesting to note that also the sublimation entropy change measured in the present work, $\Delta_{sub}S°(1050\text{ K}) = (145 \pm 6)\ J\cdot K^{-1}\cdot mol^{-1}$, disagrees with that calculated from the absolute entropy values for gaseous and solid CdF_2 selected in the database[6] at 1050 K (165 $J\cdot K^{-1}\cdot mol^{-1}$). On this basis, giving more weight to the second-law result, we propose as the standard sublimation enthalpy of CdF_2 the value of 285 $kJ\cdot mol^{-1}$ with an overestimated error of 10 $kJ\cdot mol^{-1}$.

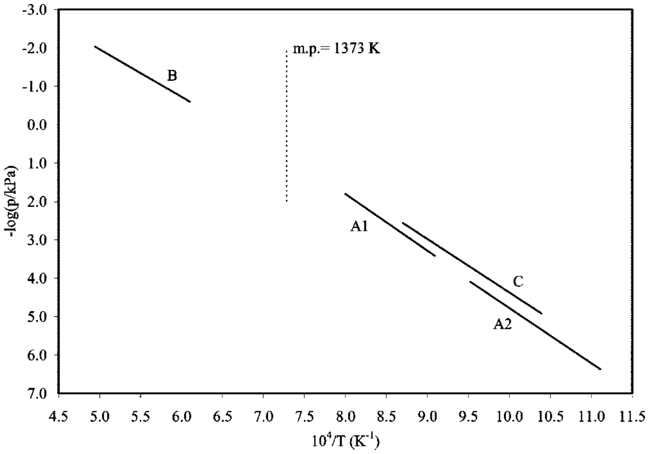

Figure 4. Comparison of the vapor pressures of CdF_2: A1 and A2 are Knudsen and Langmuir data,[2] respectively; B, boiling point data;[1] C, this work.

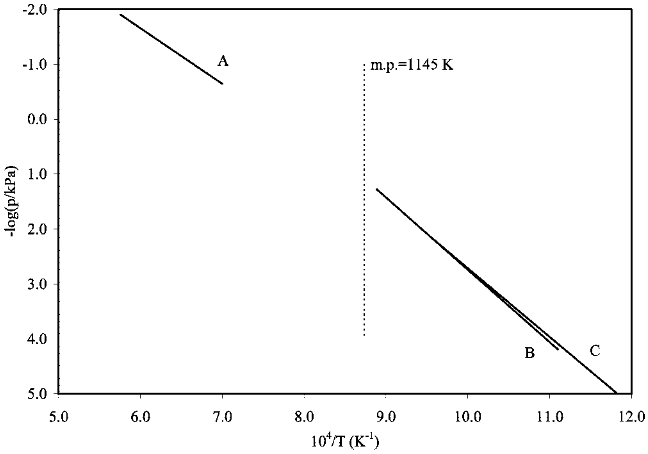

Figure 5. Comparison of the vapor pressure of ZnF_2. A, boiling point data;[1] B, Knudsen data;[3] C, this work.

B. ZnF_2. The selected eq 2 reported for comparison in Table 4 and Figure 5 is in excellent agreement, though with a small minor slope difference, with that obtained by the Knudsen method.[3] From the slope and intercept of this equation, the second-law thermodynamic changes associated with the congruent sublimation of the compound were calculated: $\Delta_{sub}H°(950\text{ K}) = (240 \pm 8)\ kJ\cdot mol^{-1}$ and $\Delta_{sub}S°(950\text{ K}) = (149 \pm 8)\ J\cdot K^{-1}\cdot mol^{-1}$, where the errors were estimated using the same considerations made for CdF_2. These values were reported at 298 K using the heat contents reported in the IVTANTHERMO database:[6] $\Delta_{sub}H°(298\text{ K}) = (252 \pm 10)\ kJ\cdot mol^{-1}$ and $\Delta_{sub}S°(298\text{ K}) = (169 \pm 8)\ J\cdot K^{-1}\cdot mol^{-1}$. Two standard sublimation enthalpy values were calculated by treating by the third-law procedure two vapor pressures of this compound at two extreme temperatures of the covered experimental ranges, (850 and 1050) K. The necessary *fef* were those reported in the database. [6] The obtained values reported in Table 5 present a very small temperature dependence. The average third-law value, $\Delta_{sub}H°(298\ K) = 262\ kJ \cdot mol^{-1}$, with an overestimated error of 4 $kJ \cdot mol^{-1}$, agrees well enough with that derived from the second-law procedure. It is necessary to remark that also for ZnF_2 the sublimation entropy change at 950 K found in the present work is lower than that selected in the database,[6] 161 $J \cdot K^{-1} \cdot mol^{-1}$. Giving equal weight to the second- and third-law results, we propose as standard sublimation enthalpy of ZnF_2 the value of (257 ± 8) $kJ \cdot mol^{-1}$.

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